

ering the entire diameter distribution of CoMoCats are approximately 1053 to 1058 kg/m³. This value range matches the stated isopycnic density of (6,5) SWNTs in Crochet et al [J. Crochet, M. Clemens, T. Hertel, *J. Am. Chem. Soc.* 2007, 129(26), 8058.] and is consistent with the unstated ρ_{SWCNT} numbers of Arnold et al. In the reported experiments, the density of the liquid, ρ_s , was set to approximately 1137 kg/m³. Thus across the entire diameter range of CoMoCats, the maximum difference in ρ_{SWCNT} was about 5 kg/m³, compared to a $\Delta\rho_i$ value of about 85 kg/m³. Thus any difference in velocity due to chirality effects was less than about 6%.

[0077] UV-Vis-NIR Spectrophotometry: UV-Vis-NIR was performed in transmission mode on a PerkinElmer Lambda 950 UV-Vis-NIR spectrophotometer over the range of 1350 to 350 nm. Measurements were typically performed on the extracted fractions in a 2 mm path length quartz cuvette. In all cases, the incident light was circularly polarized prior to the sample compartment, and the spectra corrected for both dark current and background. Data was recorded at 1 nm increments with an instrument integration time of at least 0.12 s per increment. The reference beam was left unobstructed, and the subtraction of the appropriate reference sample was performed during data reduction.

[0078] Atomic Force Microscopy: Tapping-mode atomic force microscopy (AFM) measurements were conducted in air using a Nanoscope IV system (Digital Instruments) operated under ambient conditions with 1-10 Ohm cm, phosphorous (n) doped silicon tips (Veeco; RTE5P5, 125 μm length; 30 μM width, normal spring constant, 40 N/m; resonance frequency, 240 kHz to 300 kHz). Length separated surfactant-coated tubes were diluted 100 \times in water (18 M Ωcm^{-1}) prior to being deposited (2 μL) onto plasma cleansed Si [1,1,1] wafers. After being allowed to dry, the entire sample was exposed to high intensity UV light for 2 h followed by 1 isopropanol and 3 water wash cycles using a solution deposition and wicking procedure to afford clear imaging conditions.

[0079] Under the centrifugation conditions described herein, nanotubes reached the top of the liquid column in less than 20 h, the time at which the solution volume was fractionated and the samples analyzed through ultraviolet-visible-near infrared spectroscopy (UV-Vis-NIR), dynamic light scattering (DLS), and atomic force microscopy (AFM). As in Arnold et al., iodixanol (5,5'-[(2-hydroxy-1-3 propanediyl)-bis(acetylamino)]bis [N,N'-bis(2,3dihydroxypropyl)-2,4,6-triiodo-1,3-benzenecarboxamide]), purchased as Opti-PrepTM, was used to generate the various density solutions. In the experiment described herein, CoMoCat process SWCNTs were used, however the technique has been repeated with similar results using both laser and HiPco process SWCNTs.

[0080] A schematic of aspects of a preferred embodiment process and specifically, spectra for several of the fractionated layers are shown in FIG. 3. When run to optimize the transient motion of the SWCNTs, spectra showing well-defined SWCNT peak features with increasing peak to baseline ratios are measured above the injection layer. No significant presence of SWCNT bundles, as would be observed by a combination of peak broadening and decrease in peak absorption relative to the baseline, was observed for deoxycholate dispersed SWCNTs. A small amount of high density impurities is seen to fractionate through the dense underlayer to the bottom of the tube. In sodium cholate dispersions, bundles are observed due to the significantly poorer stability of individual

SWCNTs in sodium cholate solutions. In the deoxycholate results, no change in the chirality distribution with the length separation is measurable, as is evidenced by the constant relative sizes (to each other) of the chirality specific absorption features in FIG. 3.

[0081] Referring to FIG. 3, a diagram is shown of the initial and final location of the CoMoCat SWCNTs, and UV-vis-NIR spectra for the indicated fractionation locations. The SWCNTs were injected in a 30% iodixanol mass fraction layer. The longer fractions display sharp SWCNT optical transitions with no evidence of significant chirality selection. Fractions at and below the injection layer have features that are smeared and red-shifted. This sort of absorption feature is indicative of bundling. The absorbance spectra below 375 nm and above 1300 nm contain contributions from the iodixanol that are difficult to subtract. These contributions negligibly affect the spectra in the (400 to 1300) nm range.

[0082] Lengths, shown in FIG. 4, are projected from the measured absorption ratio of the 984 nm peak to an approximate baseline value measured at 775 nm. The length dependence based on this ratio was calculated from a linear fit to the same ratio versus length measured for DNA-dispersed CoMoCat SWCNTs length separated by size exclusion chromatography. An approximate relation for the length for this batch of CoMoCat SWCNTs is:

$$\ell(\text{nm}) \approx \left(\frac{\text{Absorbance}(984 \text{ nm})}{\text{Absorbance}(775 \text{ nm})} - 0.842 \right) * 160.4 \text{ nm} \quad (6)$$

Specifically, FIG. 4 illustrates apparent length versus fraction number at 4 h, 9 h and 19 h of centrifugation. The peak height to baseline ratio for the same SWCNT material wrapped with DNA and length separated by size exclusion chromatography was also used to project the length of the nanotubes. Each fraction was 0.45 cm tall. Error bars represent 10% of the projected length value due to uncertainty in the slope of equation (6). A change to this slope affects all projected values by a uniform multiplier. The crossed circles are AFM measured values for 20 h fractions. As overlapping tubes are not counted, AFM may underestimate the actual average length. Note that the concentration is not uniform across the fractions, and that the initial distribution of lengths is centered at approximately 215 nm.

[0083] Values for the lengths measured using depolarized dynamic light scattering are in general agreement with the projected length values from the absorption. The large amount of iodixanol present in each fraction causes AFM measurement to be difficult. AFM measurements on the longest fraction isolated, and the corresponding absorption spectra (both shown in FIG. 5), yield lengths of (960 \pm 35) nm based on 175 SWNTs and 1093 nm respectively, indicating that length extrapolation using the UV-Vis-NIR absorption is sufficiently accurate. Of note is that the peak to baseline ratio shown in FIG. 5 is equivalent to the ratio calculable for the (6,5) purified samples shown in Arnold et al. Chirality purification of the sample shown in FIG. 5 would thus yield spectra with substantially larger peak to baseline values than previously demonstrated in a bulk sample. Error bars for lengths calculated by equation (5) are set to 10% of the average value, to project the estimated 10% uncertainty in the slope value.

[0084] Specifically, FIG. 5 shows the spectra of the longest separated material has a peak height to baseline ratio of